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(54) **Silicone polyether stabilized silicone latex solvent thickening**

(57) The viscosity of a solvent is modified by thickening the solvent with a silicone latex. A silicone latex having a plurality of crosslinked polysiloxane particles is first prepared by mixing the siloxane polymer, a surfactant and water; emulsifying the mixture to a gel phase; diluting the emulsion with water; adding a cure package (i.e., a catalyst, a crosslinker or both, or a self catalytic crosslinker); and then without removing the water from the latex and after the particles of siloxane polymer

in the latex have been cured, mixing the latex and solvent to thicken the solvent, forming viscous liquids, gels, and pastes. Water in the latex thickened solvent composition is stabilized by adding a silicone polyether during mixing of the latex and the solvent. These stabilized latex thickened solvent compositions have beneficial properties such as clarity, shelf stability, and ease of preparation; and therefore have wide areas of application, especially as additives in antiperspirants, deodorants and other personal care applications.

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## Description

Our invention provides improved stabilized aqueous crosslinked polysiloxane dispersions blended with silicone or organic fluids producing new materials with novel properties.

Cured silicone rubber powders have been made by first preparing a water-based emulsion of a curable silicone composition, then curing the composition to form a water based dispersion of cured silicone rubber powder by heating it to 80°C. to effect crosslinking and, finally, removing the water from the water-based dispersion to harvest the rubber. The disadvantage of this approach is that the initial water-based dispersion only contains 35% silicone solids. The remaining 65% of water is removed through a spray drying process. Agglomeration occurs in the spray drying process resulting in rubber powder particle sizes larger than those occurring in the latex (the dispersion of the crosslinked rubber). A sieving procedure is necessary to remove larger particle sizes.

Our invention provides viscosity modification of fluids by a silicone latex without removing the water from the silicone latex. This eliminates the heating, harvesting and sieving procedures of the prior art methods. The silicone latex of our invention provides an extremely high internal phase ratio latex, that is, the crosslinked or cured rubber is present up to 95%, with the remainder being the water continuous phase. Unexpectedly, the individual silicone latex particles swell with a solvent in the presence of a water continuous phase.

Our silicone latex compositions represent several significant advances in the art. First, the process of making the dispersion is improved since the high solids gel phase provides for a higher shear and lower particle size distribution. Second, because of the high polymer solids content of the diluted gel (above 75%), the composition does not require thickeners or other rheology modifiers to achieve excellent handling characteristics. A third advantage is the versatility of the process, allowing mixing of silicone polymer, water, surfactant and cure package (i.e., a catalyst, a crosslinker or both, or a self catalytic crosslinker), in the manufacture of a high solids oil-in-water emulsion as a gel phase intermediate. The high solids gel is diluted with water to form a dispersion having greater than 75% silicone solids content. The gel is further processed by adding optional ingredients, if desired, and adjusted by dilution to the desired solids content.

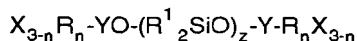
Our invention provides an improved method of modifying the viscosity of a solvent by thickening the solvent with a silicone latex which has a plurality of crosslinked polysiloxane particles. The silicone latex is prepared by (A) forming a mixture containing a siloxane polymer, a surfactant, and water; (B) emulsifying the mixture into a gel phase; (C) diluting the emulsion with further water; and (D) adding a cure package. The addition of a silicone polyether while mixing the silicone latex and a solvent, stabilizes water present in these compositions.

The cure package is a catalyst or a crosslinker or both, or a self catalytic crosslinker. Then, without removing the water from the latex and after the particles of siloxane polymer in the latex have been cured, the latex is mixed with the solvent to thicken the solvent to a desired viscosity ranging from viscous liquids to gels or pastes.

Our invention also provides improved and stabilized silicone latex solvent thickened compositions prepared according to the above method, such compositions have a variety of uses in the personal care arena, as well as other areas of application. Thus, the silicone latex provides a means to modify the rheology of various fluids which enhances formulating capability and it functions as a reservoir for various fluids and solvents.

In preparing our improved latex thickened solvent compositions, we make use of silicone latexes and methods of producing same described in the two copending applications EP 96302661.2, filed April 17, 1996, and EP 96302659.6, filed April 17, 1995 and corresponds to US Patent 5,674,937. The silicone latex is free of rheology modifiers, has a plurality of crosslinked polysiloxane particles and has at least 75% silicone content by weight. It is an aqueous dispersion of crosslinked polysiloxane capable of crosslinking via condensation, addition or free radical reactions. The latex is prepared by combining:

- (i) 100 weight parts of a siloxane polymer or polymer mixture having a viscosity of greater than 5,000 mm<sup>2</sup>/s but less than 500,000 mm<sup>2</sup>/s at 25°C., the siloxane polymer or polymer mixture having at least one polymer species of the formula:



where n is 0, 1, 2 or 3; z is an integer from 500-5,000; X is hydrogen, a vinyl group, a hydroxyl group, any condensable or hydrolyzable group; Y is a silicon atom, =Si-(CH<sub>2</sub>)<sub>m</sub>-SiR<sup>1</sup><sub>2</sub>- group, or =Si-(CH<sub>2</sub>)<sub>m</sub>-SiR<sup>1</sup><sub>2</sub>OSiR<sup>1</sup><sub>2</sub>(CH<sub>2</sub>)<sub>m</sub>-SiR<sup>1</sup><sub>2</sub>- group, where m is 1-8; R is individually selected from aliphatic alkyl, aminoalkyl, polyaminoalkyl, epoxy-alkyl, alkenyl or aromatic groups; and R<sup>1</sup> is individually selected from X, aliphatic alkyl, alkenyl and aromatic groups,

- (ii) 0.5-10 weight parts surfactant,  
(iii) 0.5-25 weight parts water,  
(iv) 0.1-20 weight parts crosslinker,

(v) 0.00001-20 weight parts catalyst, or

(vi) 1-5 weight parts of a self catalytic crosslinker in place of crosslinker (iv) and catalyst (v).

If desired, a preservative may also be included. Some examples of representative preservatives which may be employed include formaldehyde; DMDM hydantoin (CTFA); 5-bromo-5-nitro-1,3-dioxane; parabens namely methyl paraben, ethyl paraben, propyl paraben and butyl paraben; sorbic acid; diazolidinyl urea; imidazolidinyl urea; iodopropynyl butyl carbamate; 5-chloro-2-methyl-4-isothiazolin-3-one; blends of isopropyl, isobutyl and n-butyl p-hydroxybenzoic acids; or mixtures of two or more such preservatives.

Examples of condensable or hydrolyzable groups appropriate for X are halogen, alkoxy, amino, ketoxime, ureido, carboxyl, sulfate, sulfate ester, cyano, isocyanate, phosphate and phosphate ester.

The aqueous dispersion is produced by mixing the silicone polymer, surfactant and water, under sufficient shear and for a sufficient period of time to obtain a high solids "oil-in-water" emulsion, capable of forming a characteristic gel phase having at least 90% polymer solids content and having particle sizes between 0.1-20 micrometer. The cure package (i.e., a catalyst, a crosslinker or both, or a self catalytic crosslinker) is added directly to the high solids gel phase or after dilution of the gel with water to the desired solids content. Alternatively, the cure package is added to the mixture prior to the emulsification into the gel phase. A high solids gel is formed first after the emulsification step and prior to dilution of the emulsion with further water. The high solids emulsion containing silicone polymer, water, surfactant and cure package is shelf-stable and may be stored as an intermediate.

The surfactant is a nonionic surfactant, cationic surfactant, anionic surfactant, amphoteric surfactant or a mixture thereof. The surfactant is present in the composition in an amount of 0.5-10 parts by weight based on 100 parts by weight of siloxane polymer, preferably 2-10 parts by weight.

Most preferred are nonionic surfactants namely polyoxyalkylene alkyl ethers, polyoxyalkylene sorbitan esters, polyoxyalkylene esters, polyoxyalkylene alkylphenyl ethers, ethoxylated amides, ethoxylated siloxanes and block copolymers of propylene oxide and ethylene oxide. Especially useful are alkyloxy-polyethyleneoxy-polypropyleneoxy-ethanols. Some representative nonionic surfactants commercially available and useful in our invention are TRITON® XL-80N, TRITON® X-405, TERGITOL® TMN-6, TERGITOL® 15-S-40, TERGITOL® 15-S-3, TERGITOL® 15-S-5 and TERGITOL® 15-S-7, produced by Union Carbide Corporation (Danbury, Connecticut); BRIJ 30 and BRIJ 35 produced by ICI CHEMICALS (Wilmington, Delaware); MAKON 10 produced by STEPAN Company (Northfield, Illinois); ETHOMID O/17 produced by AKZO Inc. (Chicago, Illinois); and PLURONIC® F38 produced by BASF Corporation (Parsippany, New Jersey).

Some additional nonionic surfactants commercially available and useful in our invention are ALKAMULS PSML-20, a product and tradename of Rhone-Poulenc (NJ), Surfactant & Specialty Division, Cranberry, New Jersey, for polyoxyethylene sorbitan (20) monolaurate; ALKAMULS PSMO-20, a product and tradename of Rhone-Poulenc (NJ), Surfactant & Specialty Division, Cranberry, New Jersey, for polyoxyethylene sorbitan (20) monooleate; RENEX 36, a product and tradename of ICI Surfactants, Wilmington, Delaware, for ethoxylated tridecyl ether; and GENAPOL® UD, a product and trademark of Hoechst Celanese Corporation, Specialty Chemical Group, Charlotte, North Carolina, for ethoxylated undecyl alcohol.

Appropriate cationic surfactants include ARQUAD T27W, ARQUAD 16-29, ARQUAD C-33, ARQUAD T50, and ETHOQUAD T/13 ACETATE, all manufactured by AKZO CHEMIE (Chicago, Illinois); and MACKALENE 216 a product of The McIntyre Group, Ltd., University Park, Illinois.

Anionic surfactants include POLYSTEP A4, A7, A11, A15, A15-30K, A16, A16-22, A18, A13, A17, B1, B3, B5, B11, B12, B19, B20, B22, B23, B24, B-25, B27, B29, and POLYSTEP C-OP3S; ALPHA-STEP ML40, and ALPHA-STEP MC48; STEPANOL MG; and BIO-TERGE AS-40, all produced by STEPAN Company (Northfield, Illinois), HOSTAPUR SAS produced by HOECHST CELANESE (Chatham, New Jersey); HAMPOSYL C30 and HAMPOSYL L30 produced by W.R. GRACE & CO. (Lexington, Massachusetts); and MIRANATE® LEC, a trademark and product of Rhone-Poulenc (NJ), Surfactant & Specialty Division, Cranberry, New Jersey. Its INCI CTFA name is SODIUM LAURETH-13 CARBOXYLATE.

Amphoteric surfactants include REWOTERIC AM TEG, REWOTERIC AM DLM-35, REWOTERIC AM B14 LS, REWOTERIC AM CAS, and REWOTERIC AM LP, all produced by SHEREX CHEMICAL CO. (Dublin, Ohio); and SCHERCOTAIN SCAB a product of Scher Chemicals, Inc., Clifton, New Jersey.

In addition to adding the surfactant to the polysiloxane, the dispersion also includes a predetermined amount of water. The water is present in the composition in an amount of 0.5-25 parts by weight based on 100 parts by weight of siloxane polymer, preferably 6-15 parts by weight.

After the mixture of siloxane polymer, surfactant and water is formed, the mixture is emulsified by mixing with sufficient shear, and for a sufficient period of time to form a high solids gel phase. Either crosslinker or catalyst or both, or the self catalytic crosslinker, (i.e., the cure package) is added to the mixture prior to or after emulsification. Mixing is preferable at a temperature of 10-70°C. If the cure package is added after the emulsification step, it is added either prior to or after diluting the gel phase with water to the desired solids content. The gel phase should have a polymer

solids content of at least 90%, preferably 90-96%. The polymer solids content in the gel phase can be as high 96-98% polymer.

Any type of mixing equipment is used to perform the emulsification step; namely, batch mixers, planetary mixers, continuous compounders such as single or multiple screw extruders, dynamic or static mixers, colloid mills, homogenizers and sonolators, or combinations thereof.

The high solids (90%) gel phase formed by emulsifying siloxane polymer, surfactant and water, is shelf-stable and capable of being stored for as many as 24 months prior to further processing.

After emulsification, the gel phase is diluted with water to achieve a silicone solids content of greater than 75%. Generally, an amount in the range of 5-30 parts by weight is added to achieve a solids content in the range of 75-98%. A more preferred solids range is 80-92% and the most preferred range is 84-90%. The high silicone solids content of the final dispersion is critical and distinguishes this latex over compositions in the prior art.

The selection and amount of crosslinker and catalyst depends on the type of silicon cure system employed to cure the composition. In general, the amount of crosslinker is 0.1-20 parts by weight per 100 parts by weight of siloxane polymer, preferably 0.1-10 parts by weight. The amount of catalyst is 0.00001-20 parts by weight per 100 parts by weight of siloxane polymer, preferably 0.00001-10 parts by weight. Condensation systems require larger amounts of catalyst while addition systems require lesser amounts. The crosslinker or catalyst is added either individually before or after emulsification, or both are added before or after emulsification.

One class of silicon cure systems appropriate to our invention involves condensation reactions, for instance, between silanol ( $\text{Si-OH}$ ) and silicon hydride ( $\text{Si-H}$ ) groups; between silanol ( $\text{Si-OH}$ ) and hydrolyzable or condensable silyl groups namely  $\text{Si-OC(O)CH}_3$ ,  $\text{Si-NR}_2$ , and  $\text{Si-ON=CR}_2$ ; between silicon hydride and hydrolyzable or condensable groups; and between two hydrolyzable or condensable groups of the same or different species.

One example of this cure system is the reaction between a siloxane polymer bearing silanol groups and a crosslinking compound bearing hydrolyzable groups directly attached to silicon atom(s). Another example of this cure system is the reaction between a siloxane polymer bearing hydrolyzable or condensable groups directly attached to silicon atom(s) and a crosslinking compound bearing silanol groups. Yet another example of this cure system is the reaction between two siloxane polymers bearing hydrolyzable or condensable groups attached directly to silicon atom(s). A further example of this cure system is the reaction between a siloxane polymer bearing hydrolyzable or condensable groups directly attached to silicon atom(s) and a siloxane polymer bearing active hydrogen atoms namely hydroxyl, ureido, mercapto or amino groups.

Catalysts for these condensation cure chemistries effect the reaction between polymer and crosslinking compound and include organometallic compounds, amino compounds, carboxylic acids, salts of amino compounds with carboxylic acids or other acids, low molecular weight polyamide resins obtained by the reaction of excess polyamines with polybasic acids, the reaction products between epoxy compounds and an excess of polyamines and noble metal compounds.

Typically, the crosslinking compound is a silane, a siloxane oligomer, a siloxane polymer, a siloxane resin, a silicon-modified organic oligomer, a silicon-modified organic polymer or a silicon-modified organic resin, each bearing a hydrolyzable or condensable group attached directly to a silicon atom.

The class of silicon cure systems most preferred in our invention, involves addition (hydrosilylation) reactions between a silicon hydride ( $\text{Si-H}$ ) group and an alkenyl group ( $-(\text{CH}_2)_n-\text{CH}=\text{CH}_2$ ) group. The silicon hydride group is attached either to the polymer or the crosslinker. The alkenyl group is attached either to the polymer or the crosslinker. If the alkenyl group is attached to the crosslinker, the crosslinker may be organic, silicon modified organic or siloxane in nature.

The number of reactive radicals on the polymer and the crosslinker determine whether a cured elastomer is obtained. An elastomeric network is formed by the addition cure, if the sum of the reactive radicals on the polymer and the reactive radicals on the crosslinker is at least five. For example, if the polymer has two alkenyl groups and the crosslinker has three silicon hydride groups, an elastomer is obtained.

The addition cure chemistry requires a catalyst to effect the reaction between polymer and crosslinking compound. Examples of suitable catalysts preferable in the addition reaction are Group VIII transition metal (noble metal) compounds. Such noble metal catalysts are described in U.S. Patent 3,923,705. A preferred platinum compound as catalyst is the reaction product of chloroplatinic acid and an organosilicon compound containing terminal aliphatic unsaturation. It is described in U.S. Patent 3,419,593. When noble metal catalysts are used, they are added in an amount from 0.00001-0.5 parts per 100 weight parts of the siloxane polymer, preferably 0.00001-0.02 parts, most preferably 0.00001-0.002 parts.

In one addition cure system useful to the invention, the siloxane polymer bearing alkenyl groups is reacted with a crosslinker, having on average at least two silicon-bonded hydrogen atoms per molecule. The reaction occurs in the presence of a hydrosilylation catalyst. The siloxane polymer has at least one alkenyl group. However, to obtain sufficient curability, the siloxane polymer should contain at least 1.1, preferably from 1.5-4 reactive alkenyl groups.

The silicon hydride crosslinker can be a hydrolyzable silicon hydride, a polymeric or oligomeric compound con-

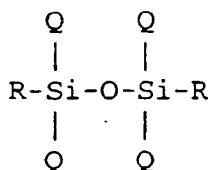
taining on average at least two hydrogen-silicon bonds such as a polyorganohydrogen siloxane, an alkylhydrogen cyclosiloxane, or a liquid copolymer containing  $\text{SiO}_2$  and/or  $\text{RSiO}_{3/2}$  units and bearing silicon-bonded hydrogen radicals. The silicon hydride crosslinker can also be an organic polymer or resin bearing Si-H groups, or a silsesquioxane containing hydrogen.

Examples of crosslinkers are trimethylsilyl endblocked polymethylhydrogen siloxane and methylhydrogen cyclosiloxane. The  $\equiv\text{SiH}$  functional crosslinker is added in sufficient amount to provide at least one hydrogen atom for each vinyl group in the polydiorganosiloxane polymer. In a typical preparation, the catalyst is present in the composition in an amount of from 0.00001-0.5 parts, and silicon hydride crosslinker in an amount of from 0.1-10 parts, each by weight, of siloxane polymer.

As previously noted, U.S. Patent No. 5,674,937 relates to a self catalytic crosslinker to perform the crosslinking function of separate catalysts and crosslinkers. In this optional mode, at least one self catalytic crosslinker is present in the composition. The term "self catalytic crosslinker" means a molecule that has at least one leaving group as the catalytic species. While in certain circumstances only one self catalytic crosslinker is needed to produce an elastomer having the desired physical properties, two or more self catalytic crosslinkers may be added to the reaction mixture. In addition, the self catalytic crosslinker or crosslinkers may be added with a conventional catalyst. However, adding the self catalytic crosslinker with a conventional catalyst is not required and the compositions may be free of conventional catalysts. The self catalytic crosslinkers are present in an amount of 1-5 parts of total self catalytic crosslinker per 100 parts by weight of siloxane polymer.

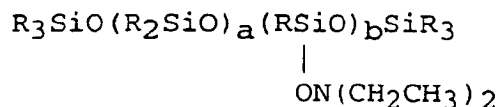
Some typical self catalytic crosslinkers include tri-functional or tetra-functional compounds namely  $\text{R-Si}(\text{Q})_3$  or  $\text{Si}(\text{Q})_4$ , where Q is carboxylic,  $-\text{OC}(\text{O})\text{R}$ , e.g. acetoxy, and R is an alkyl or alkenyl group of 1-8 carbon atoms, preferably methyl, ethyl or vinyl. Other preferred Q groups are hydroxyl amines  $-\text{ON}(\text{R})_2$ , where R is the same or different alkyl group of 1-8 carbon atoms, e.g.,  $-\text{ON}(\text{CH}_2\text{CH}_3)_2$ . Q may also be an oxime group such as  $-\text{O-N}=\text{C}(\text{R})_2$  where R is the same or different alkyl group of 1-8 carbon atoms, e.g.,  $-\text{O-N}=\text{C}(\text{CH}_3)(\text{CH}_2\text{CH}_3)$ . Further, Q is an amine group such as  $-\text{N}(\text{R})_2$  where R is the same or different alkyl group of 1-8 carbon atoms or a cyclic alkyl group, e.g.,  $-\text{N}(\text{CH}_3)_2$  or  $-\text{NH}$  (cyclohexyl). Finally, Q may be an acetamido group  $-\text{NRC}(\text{O})\text{R}$  where R is the same or different alkyl group of 1-8 carbon atoms, e.g.  $-\text{N}(\text{CH}_3)\text{C}(\text{O})\text{CH}_3$ .

In addition, partial hydrolysis products of the aforementioned compounds also function as self catalytic crosslinkers. This would include dimers, trimers and tetramers, namely oligomers of the formula:



where Q and R are as defined above.

Also useful as self catalytic crosslinkers are polymeric or copolymeric species containing 3 or more Q sites located at either pendant or terminal positions or both on the backbone of a polydiorganosiloxane molecule. Examples of the pendant group include compositions of the formula:



where R is the same or different alkyl group of 1-8 carbon atoms, a is zero or a positive integer, and b is an integer greater than two. In general, polymeric compositions having either pendant or terminal Q groups may be used, in particular compounds of the formula  $\text{Q}_{3-n}\text{R}_n\text{SiO}(\text{R}_2\text{SiO})_z\text{SiR}_n\text{Q}_{3-n}$  where n is 0, 1, 2 or 3; z is a positive integer; R is Q or independently the same or different alkyl chain of 1-8 carbon atoms, provided there are at least three Q groups in the molecule; and Q is as earlier defined.

Effective self catalytic crosslinkers are compounds which form tack free elastomers when mixed with functional silicone polymers in the absence of additional catalysts namely tin carboxylates or amines. In the self catalytic crosslinkers, the acetoxy, oxime, hydroxyl amine (aminoxyl), acetamide, and amide groups catalyze the formation of  $\equiv\text{Si-O-Si}\equiv$  bonds in reactions contemplated herein.

To prepare compositions thickened with the silicone latex, the latex is mixed with a solvent. The silicone latex is

used to thicken a single solvent or a mixture of solvents. The term solvent as used herein is intended to mean (i) organic compounds, (ii) compounds having a silicon atom, (iii) mixtures of organic compounds, (iv) mixtures of compounds having a silicon atom, or (v) mixtures of organic compounds and compounds having a silicon atom; that are used on an industrial scale to dissolve, suspend, or change the physical properties of other materials. Generally, the organic compounds are aromatic hydrocarbons, aliphatic hydrocarbons, alcohols, aldehydes, ketones, amines, esters, ethers, glycols, glycol ethers, alkyl halides or aromatic halides.

Representative of some common organic solvents are alcohols namely methanol, ethanol, 1-propanol, cyclohexanol, benzyl alcohol, 2-octanol, ethylene glycol, propylene glycol and glycerol; aliphatic hydrocarbons; namely, pentane, cyclohexane, heptane, VM&P solvent, and mineral spirits; alkyl halides namely chloroform, carbon tetrachloride, perchloroethylene, ethyl chloride and chlorobenzene; amines namely isopropylamine, cyclohexylamine, ethanolamine and diethanolamine; aromatic hydrocarbons namely benzene, toluene, ethylbenzene and xylene; esters; namely, ethyl acetate, isopropyl acetate, ethyl acetoacetate, amyl acetate, isobutyl isobutyrate and benzyl acetate; ethers namely ethyl ether, n-butyl ether, tetrahydrofuran and 1,4-dioxane; glycol ethers; namely, ethylene glycol monomethyl ether, ethylene glycol monomethyl ether acetate, diethylene glycol monobutyl ether and propylene glycol monophenyl ether; ketones namely acetone, methyl ethyl ketone, cyclohexanone, diacetone alcohol, methyl amyl ketone and diisobutyl ketone; petroleum hydrocarbons; namely, mineral oil, gasoline, naphtha, kerosene, gas oil, heavy oil and crude oil; lubricating oils; namely, spindle oil and turbine oil; fatty oils; namely, corn oil, soybean oil, olive oil, rape seed oil, cotton seed oil, sardine oil, herring oil and whale oil; and "other" miscellaneous types of organic solvents; namely, acetonitrile, nitromethane, dimethylformamide, propylene oxide, trioctyl phosphate, butyrolactone, furfural, pine oil, turpentine and m-cresol.

Representative of solvents having a silicon atom are (i) low molecular weight linear and cyclic volatile methyl siloxanes, (ii) low molecular weight linear and cyclic volatile and non-volatile alkyl and aryl siloxanes, and (iii) low molecular weight functional linear and cyclic siloxanes. Most preferred, however, are low molecular weight linear and cyclic volatile methyl siloxanes (VMS).

VMS compounds correspond to the average unit formula  $(\text{CH}_3)_a\text{SiO}_{(4-a)/2}$  in which a has an average value of two to three. The compounds contain siloxane units joined by  $\text{Si-O-Si}$  bonds. Representative units are monofunctional "M" units  $(\text{CH}_3)_3\text{SiO}_{1/2}$  and difunctional "D" units  $(\text{CH}_3)_2\text{SiO}_{2/2}$ .

The presence of trifunctional "T" units  $\text{CH}_3\text{SiO}_{3/2}$  results in the formation of branched linear or cyclic volatile methyl siloxanes. The presence of tetrafunctional "Q" units  $\text{SiO}_{4/2}$  results in the formation of branched linear or cyclic volatile methyl siloxanes.

Linear VMS have the formula  $(\text{CH}_3)_3\text{SiO}[(\text{CH}_3)_2\text{SiO}]_y\text{Si}(\text{CH}_3)_3$ . The value of y is 0-5. Cyclic VMS have the formula  $\{(\text{CH}_3)_2\text{SiO}\}_z$ . The value of z is 3-9. Preferably, these volatile methyl siloxanes have boiling points less than  $250^\circ\text{C}$ . and viscosities of 0.65 to  $5.0 \text{ mm}^2/\text{s}$ .

Representative linear volatile methyl siloxanes are hexamethyldisiloxane (MM) with a boiling point of  $100^\circ\text{C}$ ., viscosity of  $0.65 \text{ mm}^2/\text{s}$ , and formula  $\text{Me}_3\text{SiOSiMe}_3$ ; octamethyltrisiloxane (MDM) with a boiling point of  $152^\circ\text{C}$ ., viscosity of  $1.04 \text{ mm}^2/\text{s}$ , and formula  $\text{Me}_3\text{SiOMe}_2\text{SiOSiMe}_3$ ; decamethyltetrasiloxane ( $\text{MD}_2\text{M}$ ) with a boiling point of  $194^\circ\text{C}$ ., viscosity of  $1.53 \text{ mm}^2/\text{s}$ , and formula  $\text{Me}_3\text{SiO}(\text{Me}_2\text{SiO})_2\text{SiMe}_3$ ; dodecamethylpentasiloxane ( $\text{MD}_3\text{M}$ ) with a boiling point of  $229^\circ\text{C}$ ., viscosity of  $2.06 \text{ mm}^2/\text{s}$ , and formula  $\text{Me}_3\text{SiO}(\text{Me}_2\text{SiO})_3\text{SiMe}_3$ ; tetradecamethylhexasiloxane ( $\text{MD}_4\text{M}$ ) with a boiling point of  $245^\circ\text{C}$ ., viscosity of  $2.63 \text{ mm}^2/\text{s}$ , and formula  $\text{Me}_3\text{SiO}(\text{Me}_2\text{SiO})_4\text{SiMe}_3$ ; and hexadecamethylheptasiloxane ( $\text{MD}_5\text{M}$ ) with a boiling point of  $270^\circ\text{C}$ ., viscosity of  $3.24 \text{ mm}^2/\text{s}$ , and formula  $\text{Me}_3\text{SiO}(\text{Me}_2\text{SiO})_5\text{SiMe}_3$ .

Representative cyclic volatile methyl siloxanes are hexamethylcyclotrisiloxane ( $\text{D}_3$ ) a solid with a boiling point of  $134^\circ\text{C}$ . and formula  $\{(\text{Me}_2\text{SiO})_3\}$ ; octamethylcyclotetrasiloxane ( $\text{D}_4$ ) with a boiling point of  $176^\circ\text{C}$ ., viscosity of  $2.3 \text{ mm}^2/\text{s}$ , and formula  $\{(\text{Me}_2\text{SiO})_4\}$ ; decamethylcyclopentasiloxane ( $\text{D}_5$ ) with a boiling point of  $210^\circ\text{C}$ ., viscosity of  $3.87 \text{ mm}^2/\text{s}$ , and formula  $\{(\text{Me}_2\text{SiO})_5\}$ ; and dodecamethylcyclohexasiloxane ( $\text{D}_6$ ) with a boiling point of  $245^\circ\text{C}$ ., viscosity of  $6.62 \text{ mm}^2/\text{s}$  and formula  $\{(\text{Me}_2\text{SiO})_6\}$ .

Representative branched volatile methyl siloxanes are heptamethyl-3-((trimethylsilyl)oxy)trisiloxane ( $\text{M}_3\text{T}$ ) with a boiling point of  $192^\circ\text{C}$ ., viscosity of  $1.57 \text{ mm}^2/\text{s}$  and formula  $\text{C}_{10}\text{H}_{30}\text{O}_3\text{Si}_4$ ; hexamethyl-3,3-bis((trimethylsilyl)oxy)trisiloxane ( $\text{M}_4\text{Q}$ ) with a boiling point of  $222^\circ\text{C}$ ., viscosity of  $2.86 \text{ mm}^2/\text{s}$ , and formula  $\text{C}_{12}\text{H}_{36}\text{O}_4\text{Si}_5$ ; and pentamethyl-((trimethylsilyl)oxy)cyclotrisiloxane ( $\text{MD}_3$ ) with the formula  $\text{C}_8\text{H}_{24}\text{O}_4\text{Si}_4$ .

Our process also includes using low molecular weight linear and cyclic volatile and non-volatile alkyl and aryl siloxanes represented respectively by formulas  $\text{R}_3\text{SiO}(\text{R}_2\text{SiO})_y\text{SiR}_3$  and  $(\text{R}_2\text{SiO})_z$ . R can be alkyl groups with 2-20 carbon atoms or aryl groups namely phenyl. The value of y is 0-80, preferably 5-20. The value of z is 3-9, preferably 4-6. These polysiloxanes have viscosities generally in the range of  $1\text{-}100 \text{ mm}^2/\text{s}$  ( $\text{mm}^2/\text{s}$ ). Polysiloxanes are used where y has a value sufficient to provide polymers with a viscosity in the range of  $100\text{-}1,000 \text{ mm}^2/\text{s}$ . Typically, y is 80-375. Illustrative of such polysiloxanes are polydimethylsiloxane, polydiethylsiloxane, polymethylethylsiloxane, polymethylphenylsiloxane and polydiphenylsiloxane.

In addition, low molecular weight functional polysiloxanes can be employed, and are represented by the formula  $\text{R}_3\text{SiO}(\text{RQSiO})_y\text{SiR}_3$  where Q is a functional group. Examples of such functional polysiloxanes containing functional

groups represented by Q are acrylamide functional siloxane fluids, acrylate functional siloxane fluids, amide functional siloxane fluids, amino functional siloxane fluids, carbinol functional siloxane fluids, carboxy functional siloxane fluids, chloroalkyl functional siloxane fluids, epoxy functional siloxane fluids, glycol functional siloxane fluids, ketal functional siloxane fluids, mercapto functional siloxane fluids, methyl ester functional siloxane fluids, perfluoro functional siloxane fluids and silanol functional siloxanes.

Our improved, stabilized, thickened solvent compositions have particular value in the personal care arena. Because of volatility characteristics of the solvent component, the thickened solvent compositions can be used alone, or blended with other cosmetic fluids, to form a variety of over-the-counter personal care products.

Thus, it is useful as a carrier in antiperspirants, deodorants and other personal care applications, since it leaves a dry feel, and does not cool the skin upon evaporation. It is lubricious and improves the properties of skin creams, skin care lotions, moisturizers, facial treatments; namely, acne or wrinkle removers, personal and facial cleansers, bath oils, perfumes, colognes, sachets, sunscreens, pre-shave and after-shave lotions, shaving soaps and shaving lathers. It can be used in hair shampoos, hair conditioners, hair sprays, mousses, permanents, depilatories and cuticle coats, to enhance gloss and drying time, and provide conditioning benefits.

In cosmetics, it functions as a leveling and spreading agent for pigments in make-ups, color cosmetics, foundations, blushes, lipsticks, eyeliners, mascaras, oil removers, color cosmetic removers and powders. It is useful as a delivery system for oil and water soluble substances such as vitamins. When incorporated into sticks, gels, lotions, aerosols and roll-ons, our thickened solvent composition imparts a dry, silky-smooth pay-out.

In addition, our improved, stabilized, thickened solvent compositions exhibit other beneficial properties; namely, (i) clarity, (ii) the ability to combine properties of water and oil in a single phase, (iii) shelf stability, and (iv) ease of preparation; making them especially useful in antiperspirants and deodorants, in perfumes as a carrier, and for hair conditioning.

These improved, stabilized, silicone latex thickened solvent compositions have uses beyond the personal care arena, including their use as a filler or insulation material for electrical cable, a soil or water barrier for in-ground stabilization, or as a replacement for epoxy materials used in coil-on-plug designs in the electronics industry.

They are useful as carriers for crosslinked silicone rubber particles, (i) allowing ease of incorporation of the particles into such silicone or organic phases as sealants, paints, coatings, greases, adhesives, antifoams and potting compounds, and (ii) for modifying rheological, physical or energy absorbing properties of such phases in either their neat or finished condition.

In addition, the improved, stabilized, silicone latex thickened solvent compositions function as a carrier for pharmaceuticals, biocides, herbicides, pesticides and other biologically active substances; and incorporate water and water-soluble substances into hydrophobic systems. Examples of some water-soluble substances are salicylic acid, glycerol, enzymes, and glycolic acid.

The improved, stabilized, silicone latex solvent thickened compositions of our invention contain 1-99 percent by weight of the silicone latex and 1-99 percent by weight of the solvent. Preferably, however, the silicone latex solvent thickened compositions contain 5-40 percent by weight of the silicone latex and 60-95 percent by weight of the solvent. Most preferably, the silicone latex solvent thickened compositions contain 5-30 percent by weight of the silicone latex and 70-95 percent by weight of the solvent. The types of silicone latex solvent thickened compositions that are possible range from viscous fluids, to gels, to pastes, depending upon the relative amounts of silicone latex and solvent used in their preparation.

The following examples illustrate the use of the silicone latex described above to thicken a solvent. In these examples, the silicone latex is used without removing water from the latex. In addition, the solvent to be thickened is added to the silicone latex after the particles of silicone polymer in the latex have been cured.

#### EXAMPLE I - Vinyl polymer/Vinyl Gum Blend Preparation

750 g of pendant vinyl containing gum consisting of 97% dimethyl, methylvinyl siloxane, dimethylvinylsiloxy-terminated copolymer [0.19% Vi, 14,240 eq wt/Vi, degree of polymerization (DP) of 9436] were mixed with 4250 g of a vinyl-endblocked siloxane fluid (450 mm<sup>2</sup>/s, 0.46% Vi, 5853 eq wt/Vi, DP of 149) in a 10 L TURELLO mixer until a homogenous viscous blend was obtained.

#### EXAMPLE II - Preparation of Crosslinked Latex

To the 5000 g blend prepared in Example I, 102.5 g of  $\text{Me}_3\text{SiO}(\text{Me}_2\text{SiO})_3(\text{MeHSiO})_5\text{SiMe}_3$  were mixed together. To this blend 333.75 g of a 45% solution of Tergitol® TMN-6, an ethoxylated trimethylnonanol nonionic surfactant, were added. This material was mixed in a 10 L TURELLO mixer until a very high solids oil in water emulsion was formed. 215 g of deionized water was added to dilute the emulsion to a 90% silicone solids emulsion. The average particle size of the emulsion particles was 732±449 nanometer (nm) as measured by a NICOMP particle size analyzer. The emul-

sion particles were crosslinked by the addition of 12.5 g of a platinum containing complex of 92% dimethylvinylsiloxy-terminated dimethylsiloxane, 7% tetramethyldivinylsiloxane and 1% 1,3-diethenyl-1,1,3,3-tetramethyldisiloxane complex. The Pt level in this mixture was 0.5% by weight.

#### 5 EXAMPLE III - Addition of Cyclic Siloxanes to the Latex

Using a HAUSCHILD laboratory centrifugal mixer, the latex prepared in Example III and a 2.6 mm<sup>2</sup>/s silicone fluid containing a mixture of 77% octamethylcyclotetrasiloxane (D4) and 23% decamethylcyclopentasiloxane (D5), were mixed together until homogenous, by incrementally adding the D4 and D5 mixture to the latex without removing water from the latex, followed by mixing. Table I shows the results of these runs.

TABLE I

Run No.	Wt. % Cyclics	Latex (g)	Cyclics (g)	No. of Increm	grams of Cyclics/Increm	Mix Time Seconds	Results
1	50	20	20	= 4	5	104	gel
2	76.7	10	32.9	= 4	8.23	104	gel
3	84.8	5	28	= 4	7	104	liquid

#### 20 EXAMPLE IV - Addition of a Linear Siloxane to the Latex

Using the same latex prepared in Example II and without removing water from the latex, 8.14 g of the latex was mixed in a HAUSCHILD laboratory centrifugal mixer with 8.61 g of a 500 mm<sup>2</sup>/s trimethylsilyl endblocked linear polydimethylsiloxane fluid. A thick gel resulted at this fluid level of 51%. An additional 8.72 g of the same siloxane fluid were added to the mixture, and a less viscous gel was observed at this increased fluid level of 68%.

#### EXAMPLE V - Latex Made From High Viscosity Vinyl Endblocked Polymer & Blending of Latex with Cyclic Siloxanes

In a WHIP-MIX laboratory mixer 40g of a vinyl endblocked polydimethylsiloxane (PDMS) (55,000 mm<sup>2</sup>/s, 0.88% Vi, 30,810 eq wt/Vi, DP of 830), and 0.17g of the  $\equiv$ -Si-H fluid described in Example II were mixed together. 2.67 g of the 45% Tergitol® TMN-6 solution used in Example II was added. The material was mixed until a high solids emulsion was obtained and then diluted to 90% silicone solids. 0.1 g of the same platinum complex used in Example II was added to crosslink the emulsion particles. To 10 g of this latex, and without removing water from the latex, 32.9 g of the same D4 and D5 mixture used in Example III were incrementally mixed into the latex using the HAUSCHILD mixer. The total mixing time was 156 seconds. The material formed a thick gel at this fluid level of 76.7%.

These examples demonstrate that thickened solvent compositions are prepared from blends of a 90% silicone solids latex and varying amounts of octamethylcyclotetrasiloxane, decamethylcyclotetrasiloxane and linear siloxanes. Depending upon the solvent to silicone latex ratio, viscous fluids, gels or pastes are formed. By using as little as 20% silicone latex solids, gels or paste-like materials are prepared. Viscous fluids are prepared at lower silicone latex solids levels.

The following examples illustrate variations of our method for preparing latex thickened solvent compositions and the use of other types of surfactants or solvent.

#### 45 EXAMPLE VI - Cationic Surfactant

In a laboratory WHIP MIX mixer, 125 g of the vinyl endblocked PDMS in Example V, and 0.54 g of the  $\equiv$ -Si-H fluid in Example II, were mixed together. To this mixture, 10.42 g of a 40% aqueous solution of the cationic surfactant MACKALENE 216 was added. MACKALENE 216 is a tradename and product of The McIntyre Group, Ltd., University Park, Illinois. Its INCI CTFA name is RICINOLEAMIDOPROPYL DIMETHYLAMINE LACTATE. The materials were mixed until a high solids emulsion was obtained. The high solids emulsion was diluted to 90% silicone solids. 0.25 g of the platinum complex in Example II was added to crosslink (i.e., cure) the emulsion particles. 5.56 g of this latex was blended in a WHIP MIX laboratory mixer with 30.71 g of the D4 and D5 mixture in Example III. The resulting composition was a dry thick gel.



## EXAMPLE VII - Anionic Surfactant &amp; Platinum Added Prior to Emulsification

In a laboratory WHIP MIX mixer, 100 g of the vinyl endblocked PDMS in Example V, 0.43 g of the  $\equiv\text{Si-H}$  fluid in Example II, and 0.25 g of the platinum complex in EXAMPLE II, were mixed together. To this mixture, 7.50 g of a 40% aqueous solution of the anionic surfactant BIO-TERGE AS-40 was added. BIO-TERGE AS-40 is a tradename and product of Stepan Company, Northfield, Illinois and is a sodium alpha olefin sulfonate. The materials were mixed until a high solids emulsion was obtained. The high solids emulsion was diluted to 90% silicone solids. 5.56 g of this latex was blended in a WHIP MIX laboratory mixer with 30.71 g of the D4 and D5 mixture in Example III. The resulting composition was a dry thick gel.

## EXAMPLE VIII - Amphoteric Surfactant

In a WHIP MIX laboratory mixer, 125 g of the vinyl endblocked PDMS in Example V, and 0.54 g of the  $\equiv\text{Si-H}$  fluid in Example II were mixed together. To this mixture, 7.5 g of a 50% aqueous solution of the amphoteric surfactant SCHERCOTAIN SCAB was added. SCHERCOTAIN SCAB is a tradename and product of Scher Chemicals, Inc., Clifton, New Jersey. Its INCI CTFA name is COCAMIDOPROPYL HYDROXY SULTAINE. The materials were mixed until a high solids emulsion was obtained. The high solids emulsion was diluted to 90% silicone solids. 0.25 g of the platinum complex in EXAMPLE II was added to crosslink the emulsion particles. 5.56 g of this latex was blended in a WHIP MIX laboratory mixer with 30.71 g of the D4 and D5 mixture in Example III. The resulting composition was a dry thick gel.

## EXAMPLE IX - Mineral Oil as Fluid

5.56 g of the latex prepared in Example VII, and 11.67 g of mineral oil, were mixed together until a homogenous, thick, smooth, white paste was formed.

The aqueous crosslinked polysiloxane dispersions blended with silicone or organic fluids prepared by the above Examples III-IX are improved by incorporating a silicone polyether (SPE) into the silicone latex solvent thickened composition. The function of the SPE is to stabilize water carried into the composition from the latex. Stabilizing the water prevents agglomeration of the water phase, which may occur under low shear conditions namely those imparted during transportation of the thickened compositions.

Various methods are used to prevent the water in the thickened composition from agglomerating. These methods include incorporating water-in-oil emulsifiers namely the SPE, oil-in-water emulsifiers, polymeric thickeners, cosolvents, adsorbents or absorbents, into the thickened composition. However, the use of a silicone polyether has been shown to be especially effective, compared to other approaches.

The silicone polyether for use herein has the formula  $\text{MD}_{10-1,000}\text{D}'_{1-100}\text{M}$ , most preferably, the formula  $\text{MD}_{300-500}\text{D}'_{2-10}\text{M}$ , where M represents monofunctional unit  $\text{R}_3\text{SiO}_{1/2}$ , D represents difunctional unit  $\text{R}_2\text{SiO}_{2/2}$ , and D' represents difunctional unit  $\text{RR}'\text{SiO}_{2/2}$ , in which R is an alkyl group containing 1-6 carbon atoms or an aryl group, and R' is an oxyalkylene containing moiety. R' groups may contain only oxyethylene (EO) units, or a combination of oxyethylene (EO) and oxypropylene (PO) units. Preferred R' groups include oxyalkylene units in the ratio  $\text{EO}_{10-100}\text{PO}_{0-100}$ , most preferably  $\text{EO}_{12-30}\text{PO}_{0-30}$ .

R' moiety typically also includes a divalent radical; namely,  $-\text{C}_m\text{H}_{2m}-$  where m is 2-8, for connecting the oxyalkylene portion of moiety R' to the siloxane backbone; as well as a terminating radical for oxyalkylene portion of moiety R'; namely, hydrogen, hydroxyl, alkyl, aryl, alkoxy or acyloxy.

One especially preferred material for stabilizing the water phase is a silicone polyether carried in a volatile cyclic siloxane. The silicone polyether is the hydrosilylation reaction product of  $\text{MD}_{394}\text{D}''_4\text{M}$  and allyloxy-(ethylene oxide)<sub>18</sub>-(propylene oxide)<sub>18</sub>-OH, where D'' is the difunctional unit  $\text{RHSiO}_{2/2}$ . This mixture of an SPE and a volatile cyclic siloxane is added during the latex solvent blending procedure.

Samples containing this material have been found to be demonstrably more stable than samples which do not contain a silicone polyether. For example, after low speed rolling, a sample without such a stabilizer forms agglomerates of water in the blend, compared to no agglomerates when an SPE is present. Other additives such as preservatives can also be present in the latex solvent blend.

Thus, our invention provides a means of stabilizing water in a latex thickened solvent material, which allows the stabilized material to be transported without forming pockets of water in the material. One advantage of our invention, therefore, is that the SPE used to stabilize the water can be added during the latex-solvent blending process. This is much simpler and less expensive than attempting to remove the water by filtration, or by other mechanical separation techniques.

Three silicone polyethers were evaluated to demonstrate the concept of our invention. Their composition is shown below:

SPE	MW	D	D'	EO/PO	Stabilizes Water
SPE I	37283	396.0	4.0	18/18	Yes
SPE II	2388	14.4	1.8	12/0	Yes
SPE III	2390	8.7	3.7	7/0	No

The amount of SPE used to stabilize water in our invention, based on weight of active materials in the latex solvent thickened composition to be stabilized, is 0.001 to 10%, preferably 0.01 to 1%, most preferably 0.05 to 0.4%. Optionally, a preservative can be included during the blending procedure.

The following additional examples are set forth to illustrate the improved and perfecting features of our invention.

#### Example X - Silicone Latex Preparation

100 parts of a vinyl-endblocked polydimethylsiloxane (55,000 mm<sup>2</sup>/s, 0.08% Vi) and 0.80 parts of an  $\equiv$ Si-H silicone fluid (MD<sub>8</sub>D''<sub>4</sub>M) were mixed. 10.92 parts of an aqueous solution containing 27.7% TRITON® XL-80N, a nonionic surfactant sold by Union Carbide Corporation, Danbury, Connecticut; 7.69 % GERMABEN® II-E preservative, a liquid containing diazolidinyl urea and parabens, sold by Sutton Laboratories, Chatham, New Jersey; and 0.96 parts of a solution containing 70% dimethyl cyclosiloxanes, and 30% of a 0.5% platinum containing mixture of 92% of a dimethylvinylsiloxy-terminated dimethylpolysiloxane, 7% tetramethyldivinylsiloxane, and 1% 1,3-diethenyl-1,1,3,3-tetramethyldisiloxane complex; were mixed with the silicones until a 90% high solids silicone emulsion was formed. The particle size of the resulting latex was 2.2 micrometer, as measured by a Coulter particle size analyzer.

#### Example XI - Latex Cyclic Blend Preparation - No SPE

11 parts of the latex prepared in Example X, and 89 parts of a 2.6 mm<sup>2</sup>/s silicone fluid containing 77% octamethylcyclotetrasiloxane and 22% decamethylcyclopentasiloxane (referred to hereinafter as CYCLICS BLEND), were mixed together for two hours in a mixing vessel having a stirrer with a cowls blade. The mixture was then exposed to high shear by homogenizing it in a Microfluidics Corporation MICROFLUIDIZER for two passes. A thick viscous material was obtained.

#### Example XII - Latex Cyclic Blend Preparation - With SPE

Three samples were prepared following the procedure described in Example XI:

Sample 1 - 11 parts latex, 88.9 parts CYCLICS BLEND, and 0.1 parts of a silicone polyether solution containing 55% of a cyclic siloxane and 45% of an SPE which was the hydrosilylation reaction product of MD<sub>394</sub>D''<sub>4</sub>M and allyloxy-(ethylene oxide)<sub>18</sub>-(propylene oxide)<sub>18</sub>-OH

Sample 2 - 11 parts latex, 88.8 parts CYCLICS BLEND, and 0.2 parts of the above SPE solution.

Sample 3 - 11 parts latex, 88.6 parts CYCLICS BLEND, and 0.4 parts of the above SPE solution.

#### Example XIII - Low Shear Stability Testing

Sample jars (volume = 59 ml) containing latex-CYCLICS BLENDS prepared following the procedures of Examples XI and XII were rotated on a laboratory mixing wheel at 15 rpm for 16 hours. The blend from Example XI had agglomerations of water. The three samples from Example XII representative of our invention did not have agglomerations of water.

#### Example XIV - Stability of Blends Containing Preservatives

Two samples were prepared according to the procedure in Example XI.

Sample A - 11 parts latex, 88.3 parts CYCLICS BLEND, 0.1 parts of the SPE solution and 0.6 parts of LIQUAPAR® Oil preservative, a blend of isopropyl, isobutyl, and n-butyl p-hydroxybenzoic acids, and a product sold by Sutton Laboratories, Chatham, New Jersey.

Sample B - 11 parts latex, 88.15 parts CYCLICS BLEND, 0.1 parts the SPE solution, and 0.75 parts of GLYCACIL® L preservative, i.e., iodopropynyl butyl carbamate, a product sold by Lonza Inc., Fair Lawn, New Jersey.

Samples A and B were stable after evaluation by the method described in Example XIII.

Examples X-XIV, therefore, demonstrate the improvement taught by our present invention for stabilizing water in latex thickened solvents, by adding a silicone polyether during the step of mixing a latex containing water and cured crosslinked polysiloxane particles, and a solvent together to thicken the solvent.

Other variations may be made in compounds, compositions, and methods described herein without departing from the essential features of our invention.

### Claims

1. A method of modifying the viscosity of a solvent, the method comprising (I) adding a silicone latex containing water and a plurality of cured crosslinked polysiloxane particles to a solvent after the particles have been cured and without removing the water from the latex; (II) mixing the latex containing the water and the cured particles, and the solvent together to thicken the solvent; and (III) stabilizing water in the latex thickened solvent by adding a silicone polyether during step (II).
2. The method of claim 1 in which the latex is prepared by a method comprising:
  - (A) mixing (i) 100 weight parts of a siloxane polymer having a viscosity of greater than 5,000 mm<sup>2</sup>/s but less than 500,000 mm<sup>2</sup>/s at 25°C., (ii) 0.5-10 weight parts of a surfactant, and (iii) 0.5-25 weight parts of water, (B) emulsifying the mixture into a gel phase having a siloxane polymer content of at least 80% by weight; (C) diluting the emulsion with further water to a siloxane polymer content of at least 75% by weight, (D) adding 0.00001-20 weight parts catalyst either before or after the emulsification, or before or after the dilution; (E) adding 0.1-20 weight parts crosslinker either before or after the emulsification, or before or after the dilution; (F) or in place of adding (D) and (E), adding 1-5 weight parts self catalytic crosslinker either before or after the emulsification, or before or after the dilution.
3. The method of claim 2 in which the siloxane polymer has the formula  $X_{3-n}R_n-YO-(R^1_2SiO)_z-Y-R_nX_{3-n}$  where n is 0, 1, 2 or 3; z is an integer from 500-5,000; X is hydrogen, a vinyl group, a hydroxyl group, a condensable or hydrolyzable group; Y is a silicon atom, the group  $\equiv Si-(CH_2)_m-SiR^1_2-$ , or the group  $\equiv Si-(CH_2)_m-SiR^1_2OSiR^1_2(CH_2)_m-SiR^1_2-$ , where m is 1-8; R is selected from the group consisting of aliphatic alkyl, aminoalkyl, polyaminoalkyl,
4. The method of any of claims 1 to 3 in which the silicone polyether has the formula  $MD_{10-1,000}D'^{1-100}M$  or the formula  $MD_{300-500}D'^{2-10}M$ , where M represents monofunctional unit  $R_3SiO_{1/2}$ , D represents difunctional unit  $R_2SiO_{2/2}$ , and D' represents difunctional unit  $RR'SiO_{2/2}$ , in which R is an alkyl group having 1-6 carbon atoms or an aryl group, and R' is an oxyalkylene moiety.
5. The method of claim 3 in which the oxyalkylene moiety R' includes an oxyalkylene unit  $EO_{10-100}PO_{0-100}$  or an oxyalkylene unit  $EO_{12-30}PO_{0-30}$  in which EO represents  $-(CH_2CH_2O)-$  and PO represents  $-(CH_2CH(CH_3)O)-$ , respectively, epoxyalkyl, alkenyl, and an aromatic group; and R<sup>1</sup> is selected from the group consisting of X, aliphatic alkyl, alkenyl, and an aromatic group,
6. A latex thickened solvent composition obtainable by the method described in any of claims 1 to 5 having 5-40 percent by weight of the silicone latex and 60-95 percent by weight of the solvent.
7. A personal care product containing the latex thickened solvent composition of claim 6 selected from the group consisting of antiperspirants, deodorants, skin creams, skin care lotions, moisturizers, acne removers, wrinkle removers, facial cleansers, bath oils, perfumes, colognes, sachets, sunscreens, pre-shave lotions, after-shave lotions, shaving soaps, shaving lathers, hair shampoos, hair conditioners, hair sprays, mousses, permanents, depilatories, cuticle coats, make-ups, color cosmetics, foundations, blushes, lipsticks, eyeliners, mascaras, oil removers, cosmetic removers, delivery systems for oil and water soluble substances, and powders; including products in the form of sticks, gels, lotions, aerosols and roll-ons.
8. A method of treating hair or skin comprising applying to the hair or skin a composition containing the latex thickened solvent composition of claim 7.
9. A product containing the latex thickened solvent composition of claim 6 and a material selected from the group consisting of crosslinked silicone rubber particles, pharmaceuticals, biocides, herbicides, pesticides, water and water-soluble substances.

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10. A method of modifying rheological, physical, or energy absorbing properties, of silicone or organic phases selected from the group consisting of sealants, paints, coatings, greases, adhesives, antifoams and potting compounds, comprising incorporating therein the latex thickened solvent composition of claim 6 containing crosslinked silicone rubber particles.
11. A method of filling or insulating an electrical cable comprising incorporating therein the latex thickened solvent composition of claim 6.
- 10 12. A method of stabilizing in-ground soil or water barriers comprising incorporating into soil the latex thickened solvent composition of claim 6.

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**(54) Silicone polyether stabilized silicone latex solvent thickening**

(57) The viscosity of a solvent is modified by thickening the solvent with a silicone latex. A silicone latex having a plurality of crosslinked polysiloxane particles is first prepared by mixing the siloxane polymer, a surfactant and water; emulsifying the mixture to a gel phase; diluting the emulsion with water; adding a cure package (i.e., a catalyst, a crosslinker or both, or a self catalytic crosslinker); and then without removing the water from the latex and after the particles of siloxane polymer

in the latex have been cured, mixing the latex and solvent to thicken the solvent, forming viscous liquids, gels, and pastes. Water in the latex thickened solvent composition is stabilized by adding a silicone polyether during mixing of the latex and the solvent. These stabilized latex thickened solvent compositions have beneficial properties such as clarity, shelf stability, and ease of preparation; and therefore have wide areas of application, especially as additives in antiperspirants, deodorants and other personal care applications.

**EP 0 893 467 A3**



European Patent  
Office

# EUROPEAN SEARCH REPORT

Application Number  
EP 98 30 5752

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The present search report has been drawn up for all claims			
Place of search BERLIN		Date of completion of the search 15 December 1998	Examiner Hoepfner, W
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons &amp; : member of the same patent family, corresponding document</p>			

**ANNEX TO THE EUROPEAN SEARCH REPORT  
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